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SUMMARY

The ignition delay times of benzene and toluene with oxygen diluted in argon were investigated over a wide range of conditions. For benzene the concentration ranges were 0.42 to 1.69 percent fuel and 3.87 to 20.3 percent oxygen. The temperature range was 1212 to 1748 K and the reflected shock pressures were 1.7 to 7.89 atm. Statistical evaluation of the benzene experiments provided an overall equation with a 2σ confidence level as follows:

$$\tau = 1.26\times10^{-15}\exp(40\ 600/RT)[C_6H_6]^{0.42}[O_2]^{-1.70}[Ar]^{0.44}\ \text{sec}$$

For toluene the concentration ranges were 0.5 to 1.5 percent fuel and 4.48 to 13.45 percent oxygen. The temperature range was 1339 to 1797 K and the reflected shock pressures were 1.95 to 8.85 atm. The overall ignition delay equation for toluene after a statistical evaluation reads

$$\tau = 5.28\times10^{-15}\exp(55\ 090/RT)[C_7H_8]^{0.55}[O_2]^{-1.38}[Ar]^{0.35}\ \text{sec}$$

Detailed experimental information is provided.

INTRODUCTION

The oxidation kinetics of benzene and toluene has been studied since the early 1970's. Despite the time that has elapsed, the number of experimental investigations has remained relatively low, and the kinetic path of the aromatic ring oxidation process has not been successfully explained.

One of the first oxidation mechanisms for benzene was proposed by Asaba (ref. 1). According to this mechanism, phenyl radicals are formed from benzene, which reacts to give biphenyl molecules, and these were supposed to be the precursors of soot. The oxidation steps that Asaba proposed were indecisive, and he assumed the formation of a phenyl peroxy radical in the process of the ring opening.

Kern (ref. 2), who investigated benzene pyrolysis with a mass spectrometer hooked to the shock tube, found neither phenyl radicals nor biphenyl radicals and molecules to be formed in the process. Although the lack of phenyl trace

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was blamed on the low sensitivity of Kern's system, the unavailability of biphenyl species seems real.

Glassman (ref. 3), who investigated the oxidation of benzene in a flow reactor, proposed a mechanism by which the benzene is transformed to five-membered-ring intermediates, such as cyclopentadiene, which continue to decompose.

This investigation presents new experimental data on ignition delay times for benzene-oxygen-argon and toluene-oxygen-argon mixtures. Although ignition delay times are crude information and nonspecific of any distinct occurrence in the oxidation kinetics, ignition delay experiments are basic, highly reproducible, and instrument independent. Because of their positive properties and despite their drawbacks, ignition delay experiments are popular among computational kineticists and are used extensively for approval of kinetic schemes.

EXPERIMENTAL PROCEDURE

A single pulse shock tube was used in the experiments. The shock tube, made of stainless steel pipe, was flattened to a 6.35- by 6.35-cm (2.5- by 2.5-in.) square tube with rounded corners. The tube was honed and polished on all inside surfaces. The dump tank was located at the end of the driver section and separated from it by an aluminum diaphragm. A second aluminum diaphragm separated the driver and the driven sections. The two diaphragms were burst by an auxiliary short shock tube. This short shock tube had a third diaphragm that was burst by a hand plunger.

The shock speed was measured over two separate intervals with three pressure transducers whose signals were fed to a dual-channel digital oscilloscope. The shock speed was measured with an accuracy of ±0.1 μsec. A quartz pressure transducer was located in the side wall, 3 mm from the end plate. This pressure transducer was connected to a second dual-channel digital oscilloscope and was used to record and measure the ignition delay time from the pressure history of the gas (fig. 1). The ignition delay time was defined as the time from the onset of the reflected shock wave to the beginning of the pressure rise at ignition.

The mixtures were prepared in stainless steel tanks at 345-kPa total pressure by using a separate manifold. The gases and vapors were measured manometrically and allowed to expand into preevacuated stainless steel tanks. High-purity argon was used to pressurize the tank to 345 kPa. The mixtures were allowed to mix for 24 hr before use. Different mixtures of high-purity helium and argon were used as driver gases. The benzene and toluene were Fisher Scientific spectroscopical grade reagent.

The reflected shock temperatures were calculated from standard conservation equations and the ideal-gas equation of state by assuming frozen chemistry. All the thermodynamic data used were taken from new compilations (refs. 4 and 5).

RESULTS

Two sets of independent experiments were performed: one with benzene and the other with toluene. Five mixtures (A to E) were used for benzene but only three (A to D) for toluene (table I). Eighty shocks were performed for benzene and 54 for toluene. The mixtures were prepared so that power dependencies could be delineated from an empirical ignition delay equation:

$$\tau = 10^{-x}\exp(+E/RT)[\text{Fuel}]^a[O_2]^b[\text{Ar}]^c \text{ sec}$$

In each of the performed shocks the mixture composition and initial properties were known. The postshock experimental properties were the reflected temperature T_5, the density ratio ρ_5/ρ_1, and the ignition delay time τ. The experiments were spread over a wide temperature range for maximum sensitivity to the determination of the so-called activation energy. An eyeballed straight-line fit was drawn through the experimental points to help in visualizing the following explanation.

In a $\log \tau$ versus $1/T_5$ graph for benzene (fig. 2) mixtures B and C show a fourfold difference in oxygen concentration. The distance between the lines is 1.1 $\log \tau$ units.

Therefore

$$\log \tau_B = a \log(1.354\% [C_6H_6]) + b \log(5.093\% [O_2]) + c \log[\text{Ar}]$$

$$\log \tau_C = a \log(1.354\% [C_6H_6]) + b \log(4\text{x}5.093\% [O_2]) + c \log[\text{Ar}]$$

Subtracting $\log \tau_B$ from $\log \tau_C$ and disregarding the small differences in argon concentrations we get

$$\Delta[\log \tau_{(C-B)}] = b \log 4$$

$$b = \Delta[\log \tau_{(C-B)}]/\log 4 = -1.1/0.6 = -1.82$$

A plot of $\log \tau$ versus $1/T_5$ was then made for series A and E of benzene to get the argon power dependence (fig. 3):

$$\log \tau_A = a \log(1.69\% [C_6H_6]) + b \log(12.675\% [O_2]) + c \log(86.635\% [\text{Ar}])$$

$$\log \tau_E = a \log(3.28\text{x}0.516\% [C_6H_6]) + b \log(3.28\text{x}3.868\% [O_2]$$

$$+ c \log(3.28\text{x}95.616\% [\text{Ar}])$$

Subtracting $\log \tau_A$ from $\log \tau_E$ we get $\Delta[\log \tau_{(E-A)}] = 0.33$. Therefore $c \cong 0.64$.

Groups A and D of benzene differed fourfold in fuel concentration (fig. 4). Here $\Delta[\log \tau_{(A-D)}] = 0.166$ and $a = 0.28$. Groups A and B of toluene also differed threefold in fuel concentration (fig. 5). Thus $a = 0.45$. For groups B and D of toluene (fig. 6) the difference between the mixtures was threefold in oxygen concentration and $\Delta[\log \tau_{(B-D)}] = 0.72$. Thus $b = -1.50$. In the same way c was found to be approximately 0.22 (fig. 7).

This discussion explains why the mixtures were chosen as they were and how the experiments were performed. We found, however, that a statistical approach to the determination of a, b, and c was more appropriate and more accurate since it could take into consideration such factors as ρ_5/ρ_1 and differences in argon concentration between the gas mixtures. These factors were neglected in the graphical presentation in figures 2 to 7. The statistical results given below point out that the absence of these factors changed the results obtained in the graphical form.

Seventy-eight experiments were used to make the overall benzene plot of log ß versus $1/T_5$ (fig. 8) for a maximum acceptable spread of 3σ. The correlation was found with a statistical Student's t program.

$$\tau = 10^{-14.9\pm0.536}\exp(40\ 600\pm1100/RT)[C_6H_6]^{0.42\pm0.05}[O_2]^{-1.74\pm0.06}[Ar]^{0.45\pm0.07}\ \text{sec}$$

Forty-nine experiments were used to make the overall toluene plot of log ß versus $1/T_5$ (fig. 9) for a maximum acceptable spread of 2σ.

$$\tau = 10^{-14.278\pm0.388}\exp(55\ 090\pm1250/RT)[C_7H_8]^{0.55\pm0.07}[O_2]^{-1.38\pm0.07}[Ar]^{0.35\pm0.08}\ \text{sec}$$

These reduced overall ignition delay equations are in good agreement with the graphical analysis presented in figures 2 to 7. The data gathered from the benzene and toluene experiments are shown in tables II and III, respectively.

CONCLUDING REMARKS

The literature contains a relatively small amount of experimental evidence on benzene and toluene ignition delay shocks. The reason for the small number of experiments is not clear. All four papers that describe ignition delay measurements are quoted in reference 6: three experiments dealt with benzene (refs. 7, 8, and 10), and three with toluene (refs. 8 to 10). The range of experimental test conditions and the results are given in table IV.

Only one of the investigations has reduced the data in a form similar to the present study. However, any comparison would be of questionable value because of different locations for the pressure transducer used to detect the ignition delay. It has been shown (ref. 11) that locating this transducer far from the end plate results in a considerable change in the detected power and temperature dependence.

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TABLE I. - EXPERIMENTAL CONDITIONS FOR IGNITION DELAY TESTS OF BENZENE AND TOLUENE MIXTURES

Mixture	Molar composition, percent			Test section pressure before shock, P_1, torr	Number of experiments	Parameters determined
	C_6H_6	O_2	Ar			
Benzene						
A	1.69	12.675	85.63	50	22	a,c
B	1.354	5.093	93.55	↓	20	b
C	1.354	20.313	78.33	↓	13	b
D	.419	12.573	87.01	↓	13	a
E	.516	3.868	95.62	164	12	c
Toluene						
	C_7H_8	O_2	A_r			
A	1.495	13.452	85.05	50	12	a,c
B	.497	13.514	85.99	50	15	a,b
C	.497	4.476	95.03	150	14	c
D	.497	4.476	95.03	50	13	b

TABLE II. - REFLECTED PARAMETERS AND RESULTS OF BENZENE SHOCKS

Mixture	Molar composition, percent		Shock number	Test section pressure before shock, P_1, torr	Density ratio, ρ_5/ρ_1	Reflected temperature, T_5, K	Ignition delay time, τ	Test section pressure after reflected shock, P_5, atm
	C_6H_6	O_2						
A	1.69	12.675	1	50	8.15	1505	65	2.69
			2	↓	7.81	1420	178	2.43
			3	↓	7.61	1372	287	2.29
			4	↓	7.47	1339	337	2.19
			5	↓	7.99	1465	130	2.57
			6	↓	7.89	1439	151	2.49
			7	↓	7.78	1411	189	2.41
			8	↓	7.29	1297	607	2.07
			9	↓	8.20	1520	80	2.73
			10	↓	8.19	1515	84	2.72
			11	↓	8.32	1550	71	2.83
			12	↓	8.45	1587	34	2.94
			13	↓	8.53	1608	35	3.01
			14	↓	7.24	1286	750	2.04
			15	↓	7.66	1382	291	2.32
			16	↓	7.27	1293	613	2.06
			17	↓	7.44	1332	490	2.17
			18	↓	7.75	1405	159	2.39
			51	↓	7.55	1358	303	2.25
			52	↓	7.76	1408	198	2.39
			53	↓	8.33	1556	41	2.84
			54	↓	8.04	1478	92	2.60
B	1.35	5.09	101	50	7.43	1586	157	2.59
			102	↓	7.30	1544	274	2.47
			103	↓	7.13	1488	477	2.33
			104	↓	7.16	1498	478	2.35
			105	51	7.40	1573	211	2.60
			106	52	7.35	1559	243	2.61
			107	53	7.49	1605	154	2.79
			108	55	7.50	1608	146	2.88
			109	50	7.89	1748	41	3.02
			110	↓	7.68	1671	95	2.81
			111	↓	7.66	1664	91	2.79
			112	↓	7.64	1657	93	2.78
			113	51	6.72	1366	1520	2.05
			114	50	6.95	1432	480	2.18
			115	↓	7.00	1448	738	2.22
			116	↓	7.04	1461	599	2.26
			117	↓	7.02	1456	599	2.24
			151	↓	7.33	1554	226	2.50
			152	↓	7.71	1684	64	2.85
			153	↓	6.90	1419	890	2.15
C	1.35	20.31	201	50	7.61	1311	185	2.19
			202	↓	7.53	1295	272	2.14
			203	↓	7.36	1258	435	2.03
			204	↓	7.14	1212	878	1.90
			205	↓	7.23	1231	743	1.95
			206	↓	7.64	1318	202	2.21
			207	↓	7.78	1348	159	2.30
			208	↓	8.15	1435	50	2.57
			209	↓	8.37	1488	30	2.73
			210	↓	7.46	1280	330	2.09
			211	↓	8.13	1429	47	2.55
			212	↓	7.89	1373	107	2.37
			213	↓	8.03	1406	79	2.48

TABLE II. - Concluded.

Mixture	Molar composition, percent		Shock number	Test section pressure before shock, P_1, torr	Density ratio, ρ_5/ρ_1	Reflected temperature, T_5, K	Ignition delay time, τ	Test section pressure after reflected shock, P_5, atm
	C_6H_6	O_2						
D	0.419	12.57	301	51	6.84	1501	47	2.27
			302	50	6.69	1450	88	2.13
			303	↓	6.51	1390	145	1.98
			304		6.37	1348	249	1.88
			305		6.21	1299	432	1.77
			306		6.11	1272	634	1.70
			307		6.09	1265	688	1.69
			308		6.28	1319	363	1.82
			309		6.44	1369	186	1.93
			310		6.54	1400	127	2.01
			311		6.74	1467	65	2.17
			312		6.92	1527	41	2.32
			313		7.01	1560	35	2.40
E	0.516	3.87	401	164	6.54	1605	61	7.55
			402	↓	6.36	1533	122	7.01
			403		6.15	1450	273	6.41
			404		6.03	1406	415	6.10
			405		5.87	1349	755	5.69
			406		5.95	1378	604	5.90
			407		6.06	1416	412	6.17
			408		6.10	1432	367	6.28
			409	168	6.24	1486	213	6.84
			410	164	6.46	1572	98	7.30
			411	164	6.64	1652	40	7.89
			412	164	6.35	1530	122	6.99

TABLE III. - REFLECTED PARAMETERS AND RESULTS OF TOLUENE SHOCKS

Mixture	Molar composition, percent		Shock number	Test section pressure before shock, P_1, torr	Density ratio, ρ_5/ρ_1	Reflected temperature, T_5, K	Ignition delay time, τ	Test section pressure after reflected shock, P_5, atm
	C_7H_8	O_2						
A	1.495	13.45	1	50	8.12	1429	356	2.54
			2	↓	7.85	1368	975	2.36
			3		7.81	1358	1030	2.33
			4		8.20	1447	328	2.60
			5		8.37	1489	185	2.73
			6		8.57	1540	100	2.89
			7		8.70	1574	62	3.00
			8		8.91	1629	42	3.18
			9		8.62	1552	80	2.94
			10		8.99	1651	29	3.26
			11		9.37	1758	8	3.61
			12		7.97	1394	596	2.44
B	0.497	13.51	101	50	7.15	1509	73	2.37
			102	53	6.95	1448	124	2.32
			103	50	6.92	1437	186	2.18
			104	↓	6.67	1363	556	1.99
			105		6.59	1339	847	1.93
			106		6.87	1424	221	2.15
			107		6.65	1358	657	1.98
			108		7.12	1501	95	2.34
			109	51	7.53	1642	20	2.74
			110	50	7.45	1615	27	2.64
			111	↓	7.36	1582	40	2.55
			112		7.33	1572	42	2.53
			113		6.94	1443	183	2.20
			114		6.83	1412	287	2.12
			115		6.76	1391	388	2.06
C	0.497	4.48	201	150	6.22	1423	563	5.82
			202	↓	6.11	1387	741	5.58
			203		6.26	1439	490	5.93
			204		6.38	1481	330	6.22
			205	151	6.48	1521	237	6.51
			206	150	6.54	1545	175	6.65
			207	150	6.68	1597	96	7.01
			208	150	6.82	1660	42	7.45
			209	151	6.83	1663	39	7.50
			210	150	6.92	1700	26	7.74
			211	↓	7.06	1765	16	8.20
			212		6.75	1630	1629	7.24
			213		6.69	1602	63	7.04
			214		6.49	1524	208	6.51
D	0.497	4.48	301	50	7.13	1797	19	2.81
			302	↓	6.60	1566	207	2.27
			303		6.43	1501	452	2.12
			304		6.27	1441	1104	1.98
			305		6.28	1446	990	1.99
			306		6.45	1509	452	2.14
			307		6.72	1618	101	2.39
			308		6.79	1646	85	2.45
			309		6.85	1669	63	2.51
			310		7.00	1736	33	2.66
			311		6.49	1524	335	2.17
			312		6.24	1430	1311	1.96
			313		6.45	1509	370	2.14

TABLE IV. - COMPARISON OF LITERATURE EXPERIMENTS

Fuel	Fuel content, percent	Oxygen content, percent	Temperature range, K	Pressure range, atm	Calculated			Reference
					X	E	b	
Benzene	0.5 to 2.75	7.25 to 19.5	1090 to 1500	5.40 to 7.08	-10.0	42.5	-1	7,8
	0.12 to 0.42	1.58 to 1.88	1438 to 2107	2.10 to 3.39	-7.62	27.2	----	10
Toluene	0.25 to 2.75	7.25 to 9.75	1230 to 1805	4.82 to 6.70	-10.22	47.3	-1	8
	0.61 to 2.44	2.74 to 18.0	1193 to 1700	2.5 to 9.7	-17.1	58.8	-1.71	9
	0.10 to 0.37	1.63 to 1.90	1470 to 2135	2.15 to 3.33	-17.26	31.4	-1.4	10

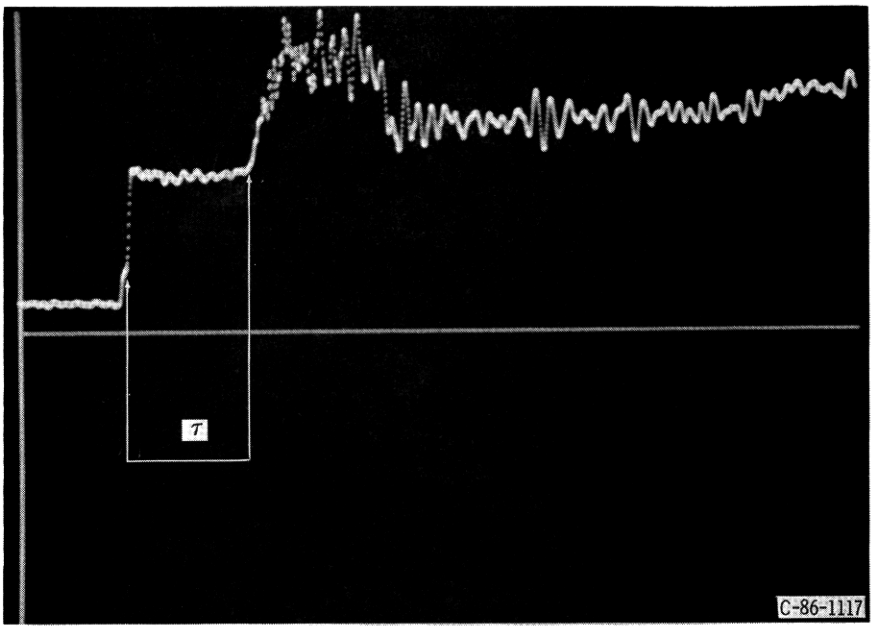

Figure 1. - Pressure history as recorded on dual-channel digital oscilloscope, with points noted for start and end of ignition delay time τ.

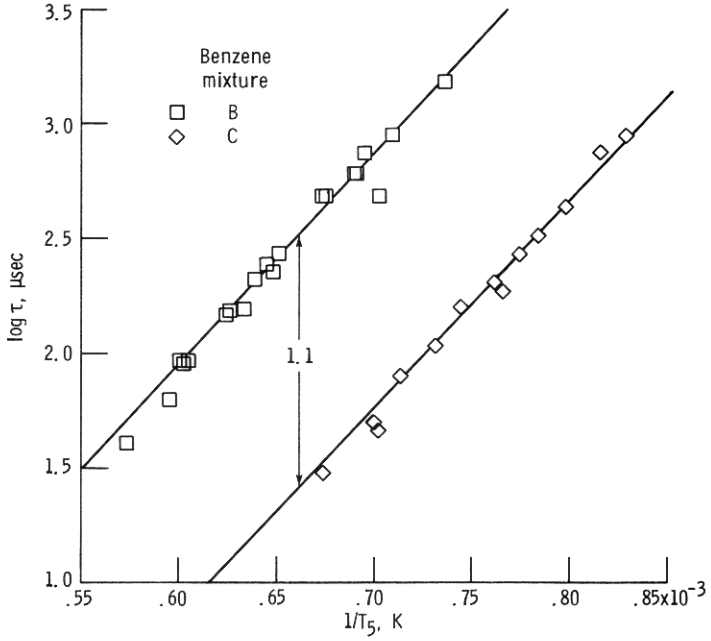

Figure 2. - Log of ignition delay time versus reciprocal reflected temperature for benzene mixtures B and C, outlining oxygen power dependence.

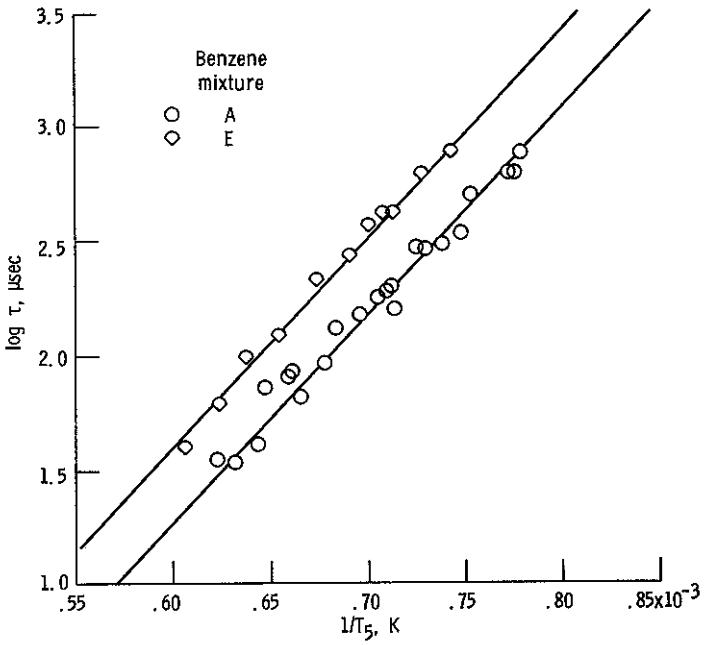

Figure 3. - Log of ignition delay time versus reciprocal reflected temperature for benzene mixtures A and E, outlining argon power dependence.

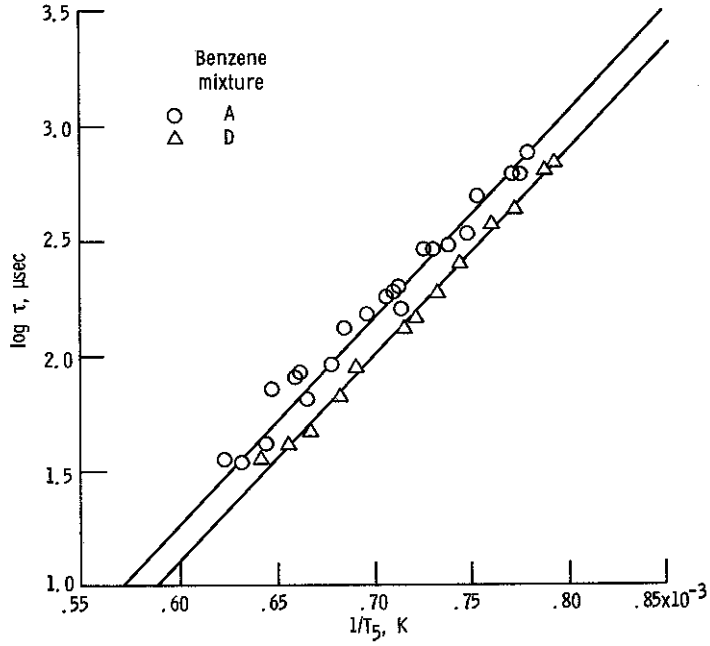

Figure 4. - Log of ignition delay time versus reciprocal reflected temperature for benzene mixtures A and D, outlining benzene power dependence.

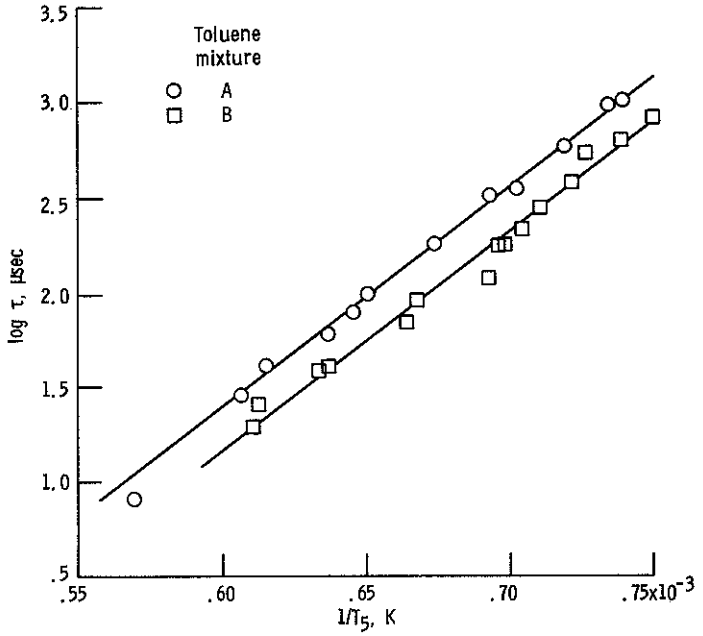

Figure 5. - Log of ignition delay time versus reciprocal reflected temperature for toluene mixtures A and B, outlining toluene power dependence.

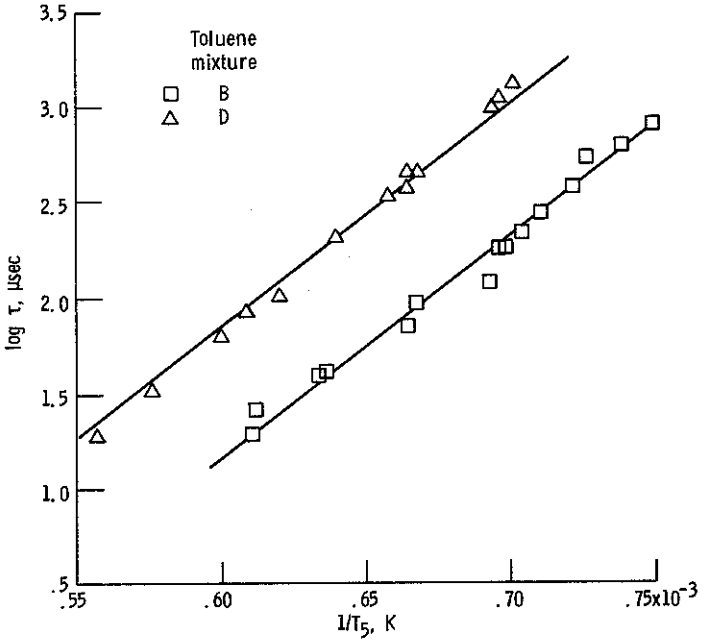

Figure 6. - Log of ignition delay time versus reciprocal reflected temperature for toluene mixtures B and D, outlining oxygen power dependence.

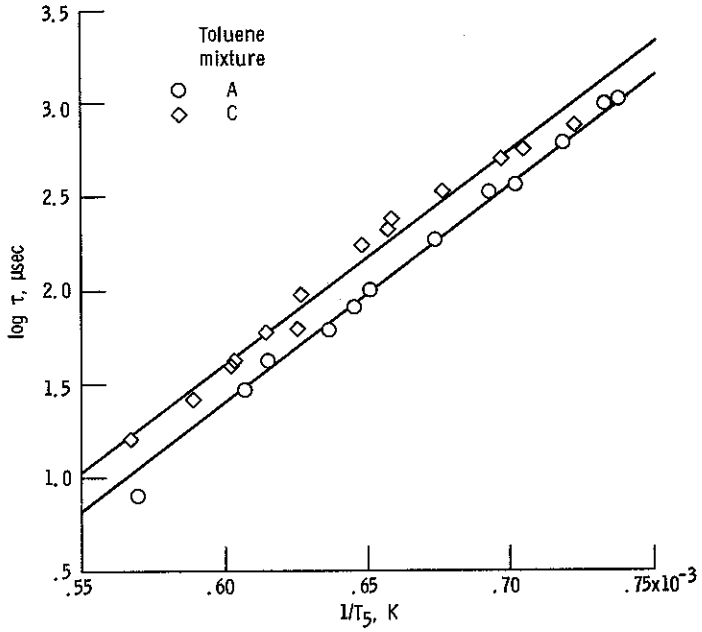

Figure 7. - Log of ignition delay time versus reciprocal reflected temperature for toluene mixtures A and C, outlining argon power dependence.

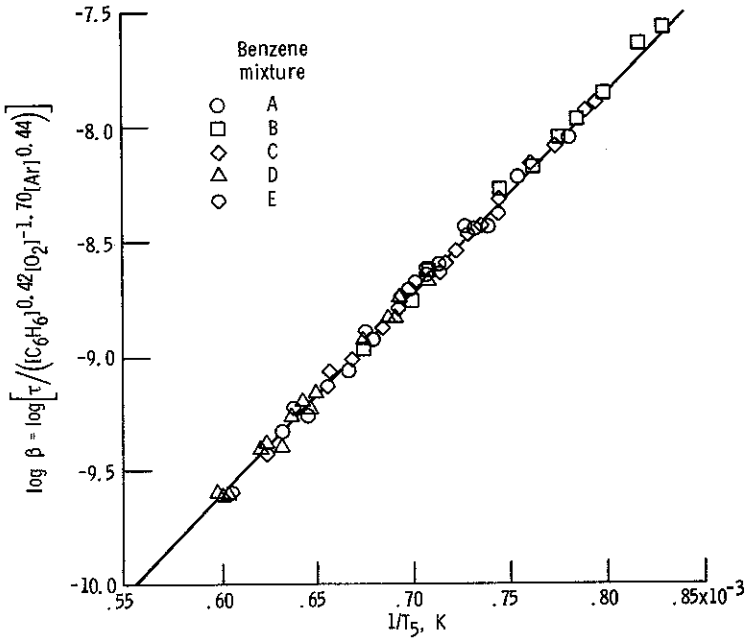

Figure 8. - Log β versus reciprocal reflected temperature for benzene experiments with a tolerated spread of 3σ. $\beta = 1.26 \times 10^{-15} \exp(40\,600/RT)$ $(cm^3)^{0.84}$ $mole^{-0.84}$ sec.

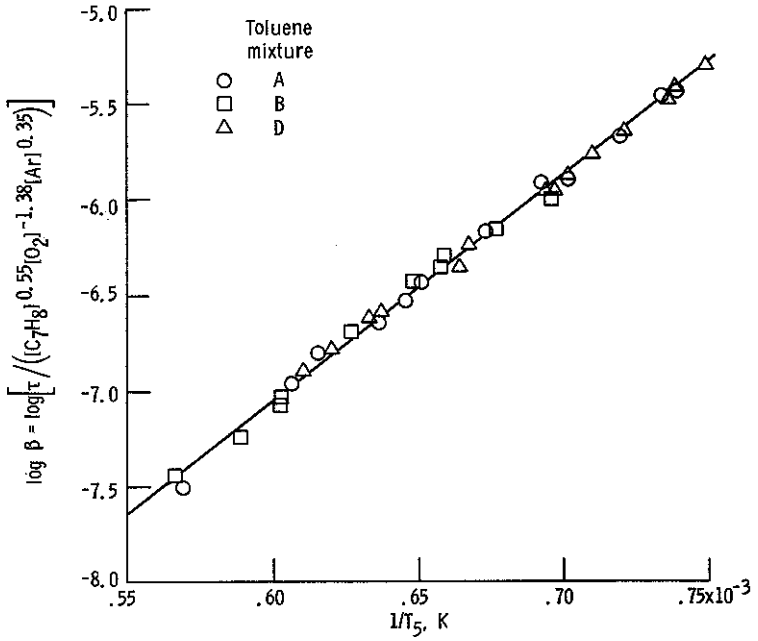

Figure 9. - Log β versus reciprocal reflected temperature for toluene experiments with a tolerated spread of 2σ. $\beta = 5.276 \times 10^{-15} \exp(55\,090/RT)$ $(cm^3)^{0.48}$ $mole^{-0.48}$ sec.

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15. Supplementary Notes

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16. Abstract

The ignition delay times of benzene and toluene with oxygen diluted in argon were investigated over a wide range of conditions. For benzene the concentration ranges were 0.42 to 1.69 percent fuel and 3.87 to 20.3 percent oxygen. The temperature range was 1212 to 1748 K and the reflected shock pressures were 1.7 to 7.89 atm. Statistical evaluation of the benzene experiments provided an overall equation with a 2σ confidence level as follows:

$$\tau = 1.26\times10^{-15}\exp(40\ 600/RT)[C_6H_6]^{0.42}[O_2]^{-1.70}[Ar]^{0.44}\ \text{sec}$$

For toluene the concentration ranges were 0.5 to 1.5 percent fuel and 4.48 to 13.45 percent oxygen. The temperature range was 1339 to 1797 K and the reflected shock pressures were 1.95 to 8.85 atm. The overall ignition delay equation for toluene after a statistical evaluation reads

$$\tau = 5.28\times10^{-15}\exp(55\ 090/RT)[C_7H_8]^{0.55}[O_2]^{-1.38}[Ar]^{0.35}\ \text{sec}$$

Detailed experimental information is provided.

17. Key Words (Suggested by Author(s))	18. Distribution Statement
Benzene; Toluene; Ignition delay time	Unclassified - unlimited STAR Category 25

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